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(54) Title: PROCESS FOR MAKING A DETERGENT COMPOSITION BY NON-TOWER PROCESS

(57) Abstract

A non-tower process for continuously preparing granular detergent composition having a density of at least about 600 g/l is provided. The process comprises the steps of: (a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer, wherein first agglomerates are formed, and (b) spraying on finely atomized liquid to the first agglomerates in a mixer, wherein second agglomerates are formed. Optionally, the second agglomerates are further subjected to the step (c), i.e., thoroughly mixing the second agglomerates in a mixer.

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**PROCESS FOR MAKING A DETERGENT COMPOSITION BY NON-TOWER
PROCESS**

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FIELD OF THE INVENTION

10 The present invention generally relates to a non-tower process for producing a particulate detergent composition. More particularly, the invention is directed to a continuous process during which detergent agglomerates are produced by feeding a surfactant and coating materials into a series of mixers. The process produces a free flowing, detergent composition whose density can be adjusted for wide range of consumer needs, and which can be commercially sold.

BACKGROUND OF THE INVENTION

20 Recently, there has been considerable interest within the detergent industry for laundry detergents which are "compact" and therefore, have low dosage volumes. To facilitate production of these so-called low dosage detergents, many attempts have been made to produce high bulk density detergents, for example with a density of 600 g/l or higher. The low dosage detergents are currently in high demand as they conserve resources and can be sold in small packages which are more convenient for consumers. However, the extent to which modern detergent products need to be "compact" in nature remains unsettled. In fact, many consumers, especially in developing countries, continue to prefer a higher dosage levels in their respective laundering operations.

30 Generally, there are two primary types of processes by which detergent granules or powders can be prepared. The first type of process involves spray-drying an aqueous detergent slurry in a spray-drying tower to produce highly porous detergent granules (e.g., tower process for low density detergent compositions). In the second type of process, the various detergent components are dry mixed after which they are agglomerated with a binder such as a

nonionic or anionic surfactant, to produce high density detergent compositions (e.g., agglomeration process for high density detergent compositions). In the above two processes, the important factors which govern the density of the resulting detergent granules are the shape, porosity and particle size distribution 5 of said granules, the density of the various starting materials, the shape of the various starting materials, and their respective chemical composition.

There have been many attempts in the art for providing processes which increase the density of detergent granules or powders. Particular attention has been given to densification of spray-dried granules by post tower treatment. For 10 example, one attempt involves a batch process in which spray-dried or granulated detergent powders containing sodium tripolyphosphate and sodium sulfate are densified and spheronized in a Marumerizer®. This apparatus comprises a substantially horizontal, roughened, rotatable table positioned within and at the base of a substantially vertical, smooth walled cylinder. This process, 15 however, is essentially a batch process and is therefore less suitable for the large scale production of detergent powders. More recently, other attempts have been made to provide continuous processes for increasing the density of "post-tower" or spray dried detergent granules. Typically, such processes require a first apparatus which pulverizes or grinds the granules and a second apparatus 20 which increases the density of the pulverized granules by agglomeration. While these processes achieve the desired increase in density by treating or densifying "post tower" or spray dried granules, they are limited in their ability to go higher in surfactant active level without subsequent coating step. In addition, treating or 25 densifying by "post tower" is not favourable in terms of economics (high capital cost) and complexity of operation. Moreover, all of the aforementioned processes are directed primarily for densifying or otherwise processing spray dried granules. Currently, the relative amounts and types of materials subjected to spray drying processes in the production of detergent granules has been limited. For example, it has been difficult to attain high levels of surfactant in the 30 resulting detergent composition, a feature which facilitates production of detergents in a more efficient manner. Thus, it would be desirable to have a process by which detergent compositions can be produced without having the limitations imposed by conventional spray drying techniques.

To that end, the art is also replete with disclosures of processes which 35 entail agglomeration of detergent compositions. For example, attempts have been

made to agglomerate detergent builders by mixing zeolite and/or layered silicates in a mixer to form free flowing agglomerates. While such attempts suggest that their process can be used to produce detergent agglomerates, they do not provide a mechanism by which starting detergent materials in the form of 5 pastes, liquids and dry materials can be effectively agglomerated into crisp, free flowing detergent agglomerates.

Accordingly, there remains a need in the art to have an agglomeration (non-tower) process for continuously producing a detergent composition having high density delivered directly from starting detergent ingredients, and preferably 10 the density can be achieved by adjusting the process condition. Also, there remains a need for such a process which is more efficient, flexible and economical to facilitate large-scale production of detergents (1) for flexibility in the ultimate density of the final composition, and (2) for flexibility in terms of incorporating several different kinds of detergent ingredients (especially liquid 15 ingredients) into the process.

The following references are directed to densifying spray-dried granules: Appel et al, U.S. Patent No. 5,133,924 (Lever); Bortolotti et al, U.S. Patent No. 5,160,657 (Lever); Johnson et al, British patent No. 1,517,713 (Unilever); and Curtis, European Patent Application 451,894.

20 The following references are directed to producing detergents by agglomeration: Beujean et al, Laid-open No.WO93/23,523 (Henkel), Lutz et al, U.S. Patent No. 4,992,079 (FMC Corporation); Porasik et al, U.S. Patent No. 4,427,417 (Korex); Beerse et al, U.S. Patent No. 5,108,646 (Procter & Gamble); Capeci et al, U.S. Patent No. 5,366,652 (Procter & Gamble); Hollingsworth et al, 25 European Patent Application 351,937 (Unilever); Swatling et al, U.S. Patent No. 5,205,958; Dhalewadikar et al, Laid Open No.WO96/04359 (Unilever).

For example, the Laid-open No.WO93/23,523 (Henkel) describes the process comprising pre-agglomeration by a low speed mixer and further 30 agglomeration step by high speed mixer for obtaining high density detergent composition with less than 25 wt % of the granules having a diameter over 2 mm. The U.S. Patent No. 4,427,417 (Korex) describes continuous process for agglomeration which reduces caking and oversized agglomerates.

None of the existing art provides all of the advantages and benefits of the present invention.

SUMMARY OF THE INVENTION

The present invention meets the aforementioned needs in the art by providing a process which produces a high density granular detergent composition. The present invention also meets the aforementioned needs in the art by providing a process which produces a granular detergent composition for flexibility in the ultimate density of the final composition from agglomeration (e.g., non-tower) process. The process does not use the conventional spray drying towers currently which is limited in producing high surfactant loading compositions. In addition, the process of the present invention is more efficient, economical and flexible with regard to the variety of detergent compositions which can be produced in the process. Moreover, the process is more amenable to environmental concerns in that it does not use spray drying towers which typically emit particulates and volatile organic compounds into the atmosphere.

As used herein, the term "agglomerates" refers to particles formed by agglomerating raw materials with binder such as surfactants and/or inorganic solutions / organic solvents and polymer solutions. As used herein, the term "mean residence time" refers to following definition:

$$\text{mean residence time (hr)} = \text{mass (kg)} / \text{flow throughput (kg/hr)}$$

All percentages used herein are expressed as "percent-by-weight" unless indicated otherwise. All ratios are weight ratios unless indicated otherwise. As used herein, "comprising" means that other steps and other ingredients which do not affect the result can be added. This term encompasses the terms "consisting of" and "consisting essentially of".

In accordance with one aspect of the invention, a process for preparing a granular detergent composition having a density at least about 600 g/l is provided.

The process comprises the steps of:

- (a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer wherein conditions of the mixer include (i) from about 2 to about 50 seconds of mean residence time, (ii) from about 4 to about 25 m/s of tip speed, and (iii) from about 0.15 to about 7 kJ/kg of energy condition wherein agglomerates are formed; and
- (b) spraying finely atomized liquid onto the agglomerates in a mixer wherein conditions of the mixer include (i) from about 0.2 to about 5

seconds of mean residence time, (ii) from about 10 to about 30 m/s of tip speed, and (iii) from about 0.15 to about 5 kJ/kg of energy condition.

Also provided is a process for preparing a granular detergent composition

5 having a density of at least about 600 g/l, the process comprises the steps of:

- (a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer wherein conditions of the mixer include (i) from about 2 to about 50 seconds of mean residence time, (ii) from about 4 to about 25 m/s of tip speed, and (iii) from about 0.15 to about 7 kJ/kg of energy condition, wherein first agglomerates are formed;
- (b) spraying finely atomized liquid onto the first agglomerates in a mixer wherein conditions of the mixer include (i) from about 0.2 to about 5 seconds of mean residence time, (ii) from about 10 to about 30 m/s of tip speed, and (iii) from about 0.15 to about 5 kJ/kg of energy condition, wherein second agglomerates are formed; and
- (c) thoroughly mixing the second agglomerates in a mixer wherein conditions of the mixer include (i) from about 0.5 to about 15 minutes of mean residence time and (ii) from about 0.15 to about 7 kJ/kg of energy condition.

Also provided are the granular detergent compositions having a high density of at least about 600g/l, produced by any one of the process embodiments described herein.

Accordingly, it is an object of the invention to provide a process for continuously producing a detergent composition which has flexibility with respect to density of the final products by controlling energy input, residence time condition, and tip speed condition in the mixers. It is also an object of the invention to provide a process which is more efficient, flexible and economical to facilitate large-scale production. These and other objects, features and attendant advantages of the present invention will become apparent to those skilled in the art from a reading of the following detailed description of the preferred embodiment and the appended claims.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

35 The present invention is directed to a process which produces free

flowing, granular detergent agglomerates having a density of at least about 600 g/l. The process produces granular detergent agglomerates from an aqueous and/or non-aqueous surfactant which is then coated with fine powder having a diameter from 0.1 to 500 microns, in order to obtain low density granules.

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Process

First Step (Step a)

In the first step of the process, one or more of aqueous and/or non-aqueous surfactant(s), which is/are in the form of powder, paste and/or liquid, and fine powder having a diameter from 0.1 to 500 microns, preferably from about 1 to about 100 microns are fed into a first mixer, so as to make agglomerates. (The definition of the surfactants and the fine powder are described in detail hereinafter.) Optionally, an internal recycle stream of powder, generally having a diameter of about 0.1 to about 300 microns, which can be generated from an "optional conditioning process (i.e., drying and/or cooling step)," which is an additional step after the process of present invention can be fed into the mixer in addition to the fine powder. The amount of such internal recycle stream of powder can be 0 to about 60 wt% of final product.

In another embodiment of the invention, the surfactant(s) can be initially fed into a mixer or pre-mixer (e.g. a conventional screw extruder or other similar mixer) prior to the above, after which the mixed detergent materials are fed into the first step mixer as described herein for agglomeration.

Generally speaking, preferably, the mean residence time of the first mixer is in range from about 2 to about 50 seconds and tip speed of the first mixer is in range from about 4 m/s to about 25 m/s, the energy per unit mass of the first mixer (energy condition) is in range from about 0.15 kj/kg to about 7 kj/kg, more preferably, the mean residence time of the first mixer is in range from about 5 to about 30 seconds and tip speed of the first mixer is in range from about 6 m/s to about 18 m/s, the energy per unit mass of the first mixer (energy condition) is in range from about 0.3 kj/kg to about 4 kj/kg, and most preferably, the mean residence time in the first mixer is in range from about 5 to about 20 seconds and tip speed of the first mixer is in range from about 8 m/s to about 18 m/s, the energy per unit mass of the first mixer (energy condition) is in range from about 0.3 kj/kg to about 4 kj/kg.

The examples of the first mixer for the first step can be any types of mixers known to persons skilled in the art, as long as the mixer can maintain the above mentioned condition for the first step. An Example can be Lödige CB Mixer manufactured by the Lödige company (Germany). As the result of the first step, 5 agglomerates having fine powder on the surface of the agglomerates (first agglomerates) are then obtained.

Second Step (Step b)

The resultant (i.e., the first agglomerates) from the first step is fed into a second mixer. Finely atomized liquid is sprayed on the agglomerates in the 10 second mixer. If excessive fine powder from the first step is optionally included in the product added to the second step, spraying the finely atomized liquid is useful in order to bind the excessive fine powder onto the agglomerates from the first step. About 0-10%, more preferably about 2-5% of powder detergent ingredients of the kind used in the first step and/or other detergent ingredients 15 can be added to the second mixer.

Generally speaking, preferably, the mean residence time of the second mixer is in range from about 0.2 to about 5 seconds and tip speed of the second mixer is in range from about 10 m/s to about 30 m/s, the energy per unit mass (energy condition) of the second mixer is in range from about 0.15 kJ/kg to about 20 5 kJ/kg, more preferably, the mean residence time of the second mixer is in range from about 0.2 to about 5 seconds and tip speed of the second mixer is in range from about 10 m/s to about 30 m/s, the energy per unit mass of the second mixer (energy condition) is in range from about 0.15 kJ/kg to about 5 kJ/kg, the most preferably, the mean residence time of the second mixer is in range from about 25 0.2 to about 5 seconds, tip speed of the second mixer is in range from about 15 m/s to about 26 m/s, the energy per unit mass of the second mixer (energy condition) is in the range from about 0.15 kJ/kg to about 2 kJ/kg.

The examples of the second mixer can be any types of mixer known to persons skilled in the art, as long as the mixer can maintain the above mentioned 30 condition for the second step. An Example can be Flexomic Model manufactured by the Schugi company (Netherlands). As the result of the second step, second agglomerates are then obtained.

Third Step [Step (c)]

If the second agglom rates are less than 600 g/l, or if further 35 agglomeration is preferred to meet the optimum condition as the final product

from th process of the present invention, the agglomerates from th second step (the second agglomerates) are fed into a third mixer. Namely, th second agglomerates are mixed and sheared thoroughly for rounding and growth of the agglomerates in the third mixer. Optionally, about 0-10% , more preferably about 5 2-5% of powder detergent ingredients of the kind used in the first step, second step, and/or other detergent ingredients can be added to the third step. Preferably, choppers which are attachable for the third mixer can be used to break up undesirable oversized agglomerates. Therefore, the process including the third mixer with choppers is useful in order to obtain reduced amount of 10 oversized agglomerates as final products, and such process is one preferred embodiment of the present invention.

Generally speaking, preferably, the mean residence time of the third mixer is in range from about 0.5 to about 15 minutes and the energy per unit mass of the third mixer (energy condition) is in range from about 0.15 to about 7 kJ/kg, 15 more preferably, the mean residence time of the third mixer is from about 3 to about 6 minutes and the energy per unit mass of the third mixer (energy condition) is in range from about 0.15 to about 4kJ/kg.

The examples of the third mixer can be any types of mixer known to persons skilled in the art, as long as the mixer can maintain the above mentioned 20 condition for the third step. An Example can be Lödige KM Mixer manufactured by the Lödige company (Germany).

As the result of the second (or the third step), a resultant product having a density of at least 600 g/l, is then obtained. Optionally, the resultant can be further subjected to drying, cooling and/or grinding.

25 In the case that the process of the present invention is proceeded by using (1) CB mixer which has flexibility to inject at least two liquid ingredients, (2) Schugi Mixer which has flexibility to inject at least two liquid ingredients, (3) KM mixer which has flexibility to inject at least a liquid ingredient, the process can incorporate five different kinds of liquid ingredients in the process. Therefore, the 30 proposed process is beneficial for persons skilled in the art in order to incorporate into a granule making process starting detergent materials which are in liquid form and are rather expensive and sometimes more difficult in terms of handling and/or storage than solid materials.

Starting Detergent Materials

The total amount of the surfactants in products made by the present invention, which are included in the following detergent materials, finely atomized liquid and adjunct detergent ingredients, is generally from about 5 % to about 60 %, more preferably from about 12% to about 40 %, more preferably, from about 15 to about 35%, in total amount of the final product obtained by the process of the present invention. The surfactants which should be included in the above can be from any part of the process of the present invention., e.g., from either one of the first step, the second step and/or the third step of the present invention.

Detergent Surfactant (Aqueous /Non-aqueous)

The amount of the surfactant of the present process can be from about 5 % to about 60 %, more preferably from about 12% to about 40 %, more preferably, from about 15 to about 35%, in total amount of the final product obtained by the process of the present invention.

The surfactant of the present process, which is used as the above mentioned starting detergent materials in the first step, is in the form of powdered, pasted or liquid raw materials.

The surfactant itself is preferably selected from anionic, nonionic, zwitterionic, amphoteric and cationic classes and compatible mixtures thereof. Detergent surfactants useful herein are described in U.S. Patent 3,664,961, Norris, issued May 23, 1972, and in U.S. Patent 3,929,678, Laughlin et al., issued December 30, 1975, both of which are incorporated herein by reference. Useful cationic surfactants also include those described in U.S. Patent 4,222,905, Cockrell, issued September 16, 1980, and in U.S. Patent 4,239,659, Murphy, issued December 16, 1980, both of which are also incorporated herein by reference. Of the surfactants, anionics and nonionics are preferred and anionics are most preferred.

Nonlimiting examples of the preferred anionic surfactants useful in the present invention include the conventional C₁₁-C₁₈ alkyl benzene sulfonates ("LAS"), primary, branched-chain and random C₁₀-C₂₀ alkyl sulfates ("AS"), the C₁₀-C₁₈ secondary (2,3) alkyl sulfates of the formula CH₃(CH₂)_x(CHOSO₃⁻M⁺) CH₃ and CH₃ (CH₂)_y(CHOSO₃⁻M⁺) CH₂CH₃ where x and (y + 1) are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing

cation, especially sodium, unsaturated sulfates such as oleyl sulfate, and the C₁₀-C₁₈ alkyl alkoxy sulfates ("AE_xS"; especially EO 1-7 ethoxy sulfates).

Useful anionic surfactants also include water-soluble salts of 2-acyloxy-alkane-1-sulfonic acids containing from about 2 to 9 carbon atoms in the acyl group and from about 9 to about 23 carbon atoms in the alkane moiety; water-soluble salts of olefin sulfonates containing from about 12 to 24 carbon atoms; and beta-alkyloxy alkane sulfonates containing from about 1 to 3 carbon atoms in the alkyl group and from about 8 to 20 carbon atoms in the alkane moiety.

Optionally, other exemplary surfactants useful in the invention include

C₁₀-C₁₈ alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C₁₀-C₁₈ glycerol ethers, the C₁₀-C₁₈ alkyl polyglycosides and the corresponding sulfated polyglycosides, and C₁₂-C₁₈ alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C₁₂-C₁₈ alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C₆-C₁₂ alkyl phenol alkoxylates (especially ethoxylates and mixed ethoxy/propoxy), C₁₀-C₁₈ amine oxides, and the like, can also be included in the overall compositions. The C₁₀-C₁₈ N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C₁₂-C₁₈ N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C₁₀-C₁₈ N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C₁₂-C₁₈ glucamides can be used for low sudsing. C₁₀-C₂₀ conventional soaps may also be used. If high sudsing is desired, the branched-chain C₁₀-C₁₆ soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

Cationic surfactants can also be used as a detergent surfactant herein and suitable quaternary ammonium surfactants are selected from mono C₆-C₁₈, preferably C₆-C₁₀ N-alkyl or alkenyl ammonium surfactants wherein remaining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl groups.

Ampholytic surfactants can also be used as a detergent surfactant herein, which include aliphatic derivatives of heterocyclic secondary and tertiary amines; zwitterionic surfactants which include derivatives of aliphatic quaternary ammonium, phosphonium and sulfonium compounds; water-soluble salts of esters of alpha-sulfonated fatty acids; alkyl ether sulfates; water-soluble salts of olefin sulfonates; beta-alkyloxy alkane sulfonates; betaines having the formula

R(R¹)₂N⁺R²COO⁻, wherein R is a C₆-C₁₈ hydrocarbyl group, preferably a C₁₀-C₁₆ alkyl group or C₁₀-C₁₆ acylamido alkyl group, each R¹ is typically C₁-C₃ alkyl, preferably methyl and R² is a C₁-C₅ hydrocarbyl group, preferably a C₁-C₃ alkylene group, more preferably a C₁-C₂ alkylene group. Examples of suitable betaines include coconut acylamidopropyldimethyl betaine; hexadecyl dimethyl betaine; C₁₂-14 acylamidopropylbetaine; C₈-14 acylamidohexyldiethyl betaine; 4[C₁₄-16 acylmethyamidodiethylammonio]-1-carboxybutane; C₁₆-18 acylamidodimethylbetaine; C₁₂-16 acylamidopentanedieethylbetaeine; and

[C₁₂-16 acylmethyamidodimethylbetaeine. Preferred betaines are C₁₂-18 dimethyl-ammonio hexanoate and the C₁₀-18 acylamidopropane (or ethane) dimethyl (or diethyl) betaines; and the sultaines having the formula (R(R¹)₂N⁺R²SO₃⁻ wherein R is a C₆-C₁₈ hydrocarbyl group, preferably a C₁₀-C₁₆ alkyl group, more preferably a C₁₂-C₁₃ alkyl group, each R¹ is typically C₁-C₃ alkyl, preferably methyl, and R² is a C₁-C₆ hydrocarbyl group, preferably a C₁-C₃ alkylene or, preferably, hydroxyalkylene group. Examples of suitable sultaines include C₁₂-C₁₄ dimethylammonio-2-hydroxypropyl sulfonate, C₁₂-C₁₄ amido propyl ammonio-2-hydroxypropyl sultaine, C₁₂-C₁₄ dihydroxyethylammonio propane sulfonate, and C₁₆-18 dimethylammonio hexane sulfonate, with C₁₂-14 amido propyl ammonio-2-hydroxypropyl sultaine being preferred.

Fine Powder

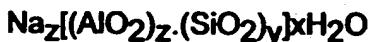
The amount of the fine powder of the present process, which is used in the first step, can be from about 94% to 30%, preferably from 86% to 54%, in total amount of starting material for the first step. The starting fine powder of the present process preferably selected from the group consisting of ground soda ash, powdered sodium tripolyphosphate (STPP), hydrated tripolyphosphate, ground sodium sulphates, aluminosilicates, crystalline layered silicates, nitrilotriacetates (NTA), phosphates, precipitated silicates, polymers, carbonates, citrates, powdered surfactants (such as powdered alkane sulfonic acids) and internal recycle stream of powder occurring from the process of the present invention, wherein the average diameter of the powder is from 0.1 to 500 microns, preferably from 1 to 300 microns, more preferably from 5 to 100 microns. In the case of using hydrated STPP as the fine powder of the present invention, STPP which is hydrated to a level of not less than 50% is preferable.

The aluminosilicate ion exchange materials used herein as a detergent builder preferably have both a high calcium ion exchange capacity and a high exchange rate. Without intending to be limited by theory, it is believed that such high calcium ion exchange rate and capacity are a function of several interrelated

- 5 factors which derive from the method by which the aluminosilicate ion exchange material is produced. In that regard, the aluminosilicate ion exchange materials used herein are preferably produced in accordance with Corkill et al, U.S. Patent No. 4,605,509 (Procter & Gamble), the disclosure of which is incorporated herein by reference.

10 Preferably, the aluminosilicate ion exchange material is in "sodium" form since the potassium and hydrogen forms of the instant aluminosilicate do not exhibit as high of an exchange rate and capacity as provided by the sodium form. Additionally, the aluminosilicate ion exchange material preferably is in over dried form so as to facilitate production of crisp detergent agglomerates as
15 described herein. The aluminosilicate ion exchange materials used herein preferably have particle size diameters which optimize their effectiveness as detergent builders. The term "particle size diameter" as used herein represents the average particle size diameter of a given aluminosilicate ion exchange material as determined by conventional analytical techniques, such as
20 microscopic determination and scanning electron microscope (SEM). The preferred particle size diameter of the aluminosilicate is from about 0.1 micron to about 10 microns, more preferably from about 0.5 microns to about 9 microns. Most preferably, the particle size diameter is from about 1 microns to about 8 microns.

- 25 Preferably, the aluminosilicate ion exchange material has the formula



wherein z and y are integers of at least 6, the molar ratio of z to y is from about 1 to about 5 and x is from about 10 to about 264. More preferably, the aluminosilicate has the formula

- 30 $\text{Na}_{12}[(\text{AlO}_2)_{12} \cdot (\text{SiO}_2)_{12}]_x \text{H}_2\text{O}$

wherein x is from about 20 to about 30, preferably about 27. These preferred aluminosilicates are available commercially, for example under designations Zeolite A, Zeolite B and Zeolite X. Alternatively, naturally-occurring or synthetically derived aluminosilicate ion exchange materials suitable for use

herein can be made as described in Krummel et al, U.S. Patent No. 3,985,669, the disclosure of which is incorporated herein by reference.

The aluminosilicates used herein are further characterized by their ion exchange capacity which is at least about 200 mg equivalent of CaCO₃ hardness/gram, calculated on an anhydrous basis, and which is preferably in a range from about 300 to 352 mg equivalent of CaCO₃ hardness/gram. Additionally, the instant aluminosilicate ion exchange materials are still further characterized by their calcium ion exchange rate which is at least about 2 grains Ca⁺⁺/gallon/minute/gram/gallon, and more preferably in a range from about 2 grains Ca⁺⁺/gallon/minute/gram/gallon to about 6 grains Ca⁺⁺/gallon/minute/gram/gallon.

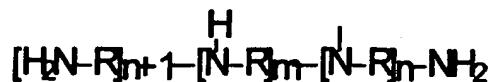
Finely Atomized Liquid

The amount of the finely atomized liquid of the present process can be from about 1 % to about 10% (active basis), preferably from 2% to about 6% (active basis) in total amount of the final product obtained by the process of the present invention. The finely atomized liquid of the present process can be selected from the group consisting of liquid silicate, anionic or cationic surfactants which are in liquid form, aqueous or non-aqueous polymer solutions, water and mixtures thereof. Other optional examples for the finely atomized liquid of the present invention can be sodium carboxy methyl cellulose solution, polyethylene glycol (PEG), and solutions of dimethylene triamine pentamethyl phosphonic acid (DETMP).

The preferable examples of the anionic surfactant solutions which can be used as the finely atomized liquid in the present inventions are about 88 - 97% active HLAS, about 30 - 50% active NaLAS, about 28% active AE3S solution, about 40-50% active liquid silicate, and so on.

Cationic surfactants can also be used as finely atomized liquid herein and suitable quaternary ammonium surfactants are selected from mono C₆-C₁₆, preferably C₆-C₁₀ N-alkyl or alkenyl ammonium surfactants wherein remaining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl groups.

Preferable examples of the aqueous or non-aqueous polymer solutions which can be used as the finely atomized liquid in the present inventions are modified polyamines which comprise a polyamine backbone corresponding to the formula:



having a modified polyamine formula $V_{(n+1)}W_mY_nZ$ or a polyamine backbone corresponding to the formula:

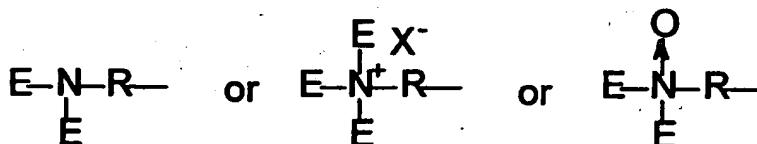
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having a modified polyamine formula $V_{(n-k+1)}W_mY_nY'_kZ$, wherein k is less than or equal to n, said polyamine backbone prior to modification has a molecular weight greater than about 200 daltons, wherein

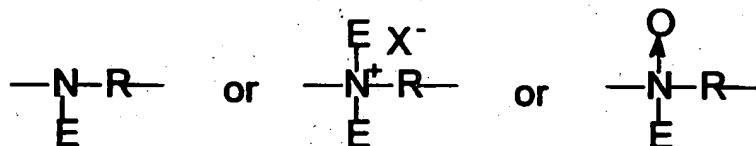
10

i) V units are terminal units having the formula:



15

ii) W units are backbone units having the formula:

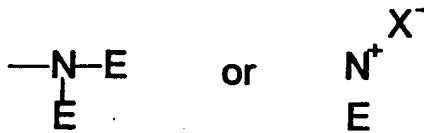


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iii) Y units are branching units having the formula:



iv) Z units are terminal units having the formula:



wherein backbone linking R units are selected from the group consisting of C₂-C₁₂ alkylene, C₄-C₁₂ alkenylene, C₃-C₁₂ hydroxyalkylene, C₄-C₁₂ dihydroxyalkylene, C₈-C₁₂ dialkylarylene, -(R¹O)_xR¹-, -(R¹O)_xR⁵(OR¹)_x-.

- 5 -(CH₂CH(OR²)CH₂O)_z(R¹O)_yR¹(OCH₂CH(OR²)CH₂)_w-,
-C(O)(R⁴), C(O)-, -CH₂CH(OR²)CH₂-; and mixtures thereof; wherein R¹ is C₂-C₆ alkylene and mixtures thereof; R² is hydrogen, -(R¹O)_xB, and mixtures thereof; R³ is C₁-C₁₈ alkyl, C₇-C₁₂ arylalkyl, C₇-C₁₂ alkyl substituted aryl, C₆-C₁₂ aryl, and mixtures thereof; R⁴ is C₁-C₁₂ alkylene, C₄-C₁₂ alkenylene, C₈-C₁₂ arylalkylene, C₆-C₁₀ arylene, and mixtures thereof; R⁵ is C₁-C₁₂ alkylene, C₃-C₁₂ hydroxyalkylene, C₄-C₁₂ dihydroxy-alkylene, C₈-C₁₂ dialkylarylene,
-C(O)-, -C(O)NHR⁶NHC(O)-, -R¹(OR¹)-, -C(O)(R⁴), C(O)-, -CH₂CH(OH)CH₂-,
-CH₂CH(OH)CH₂O(R¹O)_yR¹OCH₂CH(OH)CH₂-; and mixtures thereof; R⁶ is C₂-C₁₂ alkylene or C₆-C₁₂ arylene; E units are selected from the group
- 10 consisting of hydrogen, C₁-C₂₂ alkyl, C₃-C₂₂ alkenyl, C₇-C₂₂ arylalkyl, C₂-C₂₂ hydroxyalkyl, -(CH₂)_pCO₂M, -(CH₂)_qSO₃M, -CH(CH₂CO₂M)CO₂M,
-(CH₂)_pPO₃M, -(R¹O)_xB, -C(O)R³, and mixtures thereof; oxide; B is hydrogen, C₁-C₆ alkyl, -(CH₂)_qSO₃M, -(CH₂)_pCO₂M, -(CH₂)_q(CHSO₃M)CH₂SO₃M,
-(CH₂)_q(CHSO₂M)CH₂SO₃M, -(CH₂)_pPO₃M, -PO₃M, and mixtures thereof; M is hydrogen or a water soluble cation in sufficient amount to satisfy charge balance; X is a water soluble anion; m has the value from 4 to about 400; n has the value from 0 to about 200; p has the value from 1 to 6, q has the value from 0 to 6; r has the value of 0 or 1; w has the value 0 or 1; x has the value from 1 to 100; y has the value from 0 to 100; z has the value 0 or 1. One example of the
- 15 most preferred polyethyleneimines would be a polyethyleneimine having a molecular weight of 1800 which is further modified by ethoxylation to a degree of approximately 7 ethyleneoxy residues per nitrogen (PEI 1800, E7). It is preferable for the above polymer solution to be pre-complex with anionic surfactant such as NaLAS.
- 20 Other preferable examples of the aqueous or non-aqueous polymer solutions which can be used as the finely atomized liquid in the present invention are polymeric polycarboxylate dispersants which can be prepared by

polymerizing or copolymerizing suitable unsaturated monomers, preferably in their acid form. Unsaturated monomeric acids that can be polymerized to form suitable polymeric polycarboxylates include acrylic acid, maleic acid (or maleic anhydride), fumaric acid, itaconic acid, aconitic acid, mesaconic acid, citraconic

5 acid and methylenemalonic acid. The presence in the polymeric polycarboxylates herein of monomeric segments, containing no carboxylate radicals such as vinylmethyl ether, styrene, ethylene, etc. is suitable provided that such segments do not constitute more than about 40% by weight of the polymer.

10 Homo-polymeric polycarboxylates which have molecular weights above 4000, such as described next are preferred. Particularly suitable homo-polymeric polycarboxylates can be derived from acrylic acid. Such acrylic acid-based polymers which are useful herein are the water-soluble salts of polymerized acrylic acid. The average molecular weight of such polymers in the acid form preferably ranges from above 4,000 to 10,000, preferably from above 15 4,000 to 7,000, and most preferably from above 4,000 to 5,000. Water-soluble salts of such acrylic acid polymers can include, for example, the alkali metal, ammonium and substituted ammonium salts.

15 Co-polymeric polycarboxylates such as Acrylic/maleic-based copolymers may also be used. Such materials include the water-soluble salts of copolymers of acrylic acid and maleic acid. The average molecular weight of such copolymers in the acid form preferably ranges from about 2,000 to 100,000, more preferably from about 5,000 to 75,000, most preferably from about 7,000 to 25 65,000. The ratio of acrylate to maleate segments in such copolymers will generally range from about 30:1 to about 1:1, more preferably from about 10:1 to 2:1. Water-soluble salts of such acrylic acid/maleic acid copolymers can include, for example, the alkali metal, ammonium and substituted ammonium salts. It is preferable for the above polymer solution to be pre-complexed with anionic surfactant such as LAS.

30 Adjunct Detergent Ingredients

The starting detergent material in the present process can include additional detergent ingredients and/or, any number of additional ingredients can be incorporated in the detergent composition during subsequent steps of the present process. These adjunct ingredients include other detergent builders, bleaches, bleach activators, suds boosters or suds suppressors, anti-tarnish and

anticorrosion agents, soil suspending agents, soil release agents, germicides, pH adjusting agents, non-builder alkalinity sources, chelating agents, smectite clays, enzymes, enzyme-stabilizing agents and perfumes. See U.S. Patent 3,936,537, issued February 3, 1976 to Baskerville, Jr. et al., incorporated herein by reference.

Other builders can be generally selected from the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, borates, polyhydroxy sulfonates, polyacetates, carboxylates, and polycarboxylates. Preferred are the alkali metal, especially sodium, salts of the above. Preferred for use herein are the phosphates, carbonates, C₁₀-18 fatty acids, polycarboxylates, and mixtures thereof. More preferred are sodium tripolyphosphate, tetrasodium pyrophosphate, citrate, tartrate mono- and di-succinates, and mixtures thereof (see below).

In comparison with amorphous sodium silicates, crystalline layered sodium silicates exhibit a clearly increased calcium and magnesium ion exchange capacity. In addition, the layered sodium silicates prefer magnesium ions over calcium ions, a feature necessary to insure that substantially all of the "hardness" is removed from the wash water. These crystalline layered sodium silicates, however, are generally more expensive than amorphous silicates as well as other builders. Accordingly, in order to provide an economically feasible laundry detergent, the proportion of crystalline layered sodium silicates used must be determined judiciously. Such crystalline layered sodium silicates are discussed in Corkill et al, U.S. Patent No. 4,605,509, previously incorporated herein by reference.

Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from about 6 to 21, and orthophosphates. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene diphosphonic acid, the sodium and potassium salts of ethane 1-hydroxy-1,1-diphosphonic acid and the sodium and potassium salts of ethane, 1,1,2-triphosphonic acid. Other phosphorus builder compounds are disclosed in U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148, all of which are incorporated herein by reference.

Examples of nonphosphorus, inorganic builders are tetraborated hydrates and silicates having a weight ratio of SiO_2 to alkali metal oxide of from about 0.5 to about 4.0, preferably from about 1.0 to about 2.4. Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates and polyhydroxy sulfonates. Examples of polyacetate and polycarboxylate builders are the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylene diamine tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric acid.

Polymeric polycarboxylate builders are set forth in U.S. Patent 3,308,067, Diehl, issued March 7, 1967, the disclosure of which is incorporated herein by reference. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylene malonic acid. Some of these materials are useful as the water-soluble anionic polymer as hereinafter described, but only if in intimate admixture with the non-soap anionic surfactant.

Other suitable polycarboxylates for use herein are the polyacetal carboxylates described in U.S. Patent 4,144,226, issued March 13, 1979 to Crutchfield et al, and U.S. Patent 4,246,495, issued March 27, 1979 to Crutchfield et al, both of which are incorporated herein by reference. These polyacetal carboxylates can be prepared by bringing together under polymerization conditions an ester of glyoxylic acid and a polymerization initiator. The resulting polyacetal carboxylate ester is then attached to chemically stable end groups to stabilize the polyacetal carboxylate against rapid depolymerization in alkaline solution, converted to the corresponding salt, and added to a detergent composition. Particularly preferred polycarboxylate builders are the ether carboxylate builder compositions comprising a combination of tartrate monosuccinate and tartrate disuccinate described in U.S. Patent 4,663,071, Bush et al., issued May 5, 1987, the disclosure of which is incorporated herein by reference.

Bleaching agents and activators are described in U.S. Patent 4,412,934, Chung et al., issued November 1, 1983, and in U.S. Patent 4,483,781, Hartman, issued November 20, 1984, both of which are incorporated herein by reference.

Chelating agents are also described in U.S. Patent 4,663,071, Bush et al., from Column 17, line 54 through Column 18, line 68, incorporated herein by reference. Suds modifiers are also optional ingredients and are described in U.S. Patents 3,933,672, issued January 20, 1976 to Bartoletta et al., and 5 4,136,045, issued January 23, 1979 to Gault et al., both incorporated herein by reference.

Suitable smectite clays for use herein are described in U.S. Patent 4,762,645, Tucker et al, issued August 9, 1988, Column 6, line 3 through Column 7, line 24, incorporated herein by reference. Suitable additional 10 detergency builders for use herein are enumerated in the Baskerville patent, Column 13, line 54 through Column 16, line 16, and in U.S. Patent 4,663,071, Bush et al, issued May 5, 1987, both incorporated herein by reference.

Optional Process Steps

15 Optionally, the process can comprise the step of spraying an additional binder in one or more than one of the first, second and/or the third mixers for the present invention. A binder is added for purposes of enhancing agglomeration by providing a "binding" or "sticking" agent for the detergent components. The binder is preferably selected from the group consisting of water, anionic 20 surfactants, nonionic surfactants, liquid silicates, polyethylene glycol, polyvinyl pyrrolidone polyacrylates, citric acid and mixtures thereof. Other suitable binder materials including those listed herein are described in Beers et al, U.S. Patent No. 5,108,646 (Procter & Gamble Co.), the disclosure of which is incorporated herein by reference.

25 Other optional steps contemplated by the present process include screening the oversized detergent agglomerates in a screening apparatus which can take a variety of forms including but not limited to conventional screens chosen for the desired particle size of the finished detergent product. Other optional steps include conditioning of the detergent agglomerates by subjecting 30 the agglomerates to additional drying by way of apparatus discussed previously.

Another optional step in the process entails finishing the resulting detergent agglomerates by a variety of processes including spraying and/or admixing other conventional detergent ingredients. For example, the finishing step encompasses spraying perfumes, brighteners and enzymes onto the

finished agglomerates to provide a more complete detergent composition. Such techniques and ingredients are well known in the art.

Another optional step in the process involves surfactant paste structuring process, e.g., hardening an aqueous anionic surfactant paste by incorporating a
5 paste-hardening material by using an extruder, prior to the process of the present invention. The details of the surfactant paste structuring process is disclosed co-application No. PCT/US96/15960 (filed October 4, 1996).

In order to make the present invention more readily understood, reference is made to the following examples, which are intended to be illustrative only and
10 not intended to be limiting in scope.

EXAMPLES

Example 1:

The following is an example for obtaining agglomerates having high
15 density, using Lödige CB mixer (CB-30), followed by Schugi FX-160 Mixer.

[Step 1] 250 - 270 kg/hr of aqueous CFAS (coconut fatty alcohol sulfate surfactant) paste (C₁₂-C₁₈, 71.5% active) is dispersed by the pin tools of a CB-30 mixer along with 220 kg/hr of powdered STPP (mean particle size of 40 - 75 microns), 160 - 200 kg/hr of ground soda ash (mean particle size of 15 microns),
20 80- 120 kg/hr of ground sodium sulfate (mean particle size of 15 microns), and the 200 kg/hr of internal recycle stream of powder. The surfactant paste is fed at about 40 to 52°C, and the powders are fed at room temperature. The condition of the CB-30 mixer is as follows:

Mean residence time : 10-18 seconds

25 Tip speed : 7.5 - 14 m/s

Energy condition : 0.5 - 4 kJ/kg

Mixer speed : 550 - 900 rpm

Jacket temperature : 30°C

[Step 2] The agglomerates from the CB-30 mixer are fed to the Schugi
30 FX-160 mixer. 30 kg/hr of HLAS (an acid precursor of C₁₁-C₁₈ alkyl benzene sulfonate; 94 - 97% active) is dispersed as finely atomized liquid in the Schugi mixer at about 50 to 60°C. 20-80 kg/hr of soda ash (mean particle size of about 10 - 20 microns) is added in the Schugi mixer. The condition of the Schugi mixer is as follows:

35 Mean residence time : 0.2 - 5 seconds

Tip speed : 16 - 26 m/s

Energy condition : 0.15 - 2 kJ/kg

Mixer speed : 2000 - 3200 rpm

The resulting granules from the step 2 have a density of about 600g/l, and can

- 5 be optionally subjected to the optional process of drying, cooling, sizing and/or grinding.

Example 2:

The following is an example for obtaining agglomerates having high density, using Lödige CB mixer (CB-30), followed by Schugi FX-160 Mixer, then
10 Lödige KM mixer (KM-600).

[Step 1] 15 kg/hr - 30kg/hr of HLAS (an acid precursor of C₁₁-C₁₈ alkyl benzene sulfonate; 95 % active) at about 50 °C, and 250 - 270 kg/hr of aqueous CFAS (coconut fatty alcohol sulfate surfactant) paste (C₁₂-C₁₈, 70 % active) is dispersed by the pin tools of a CB-30 mixer along with 220 kg/hr of powdered
15 STPP (mean particle size of 40 - 75 microns), 160 - 200 kg/hr of ground soda ash (mean particle size of 15 microns), 80- 120 kg/hr of ground sodium sulfate (mean particle size of 15 microns), and the 200 kg/hr of internal recycle stream of powder. The surfactant paste is fed at about 45 to 52°C, and the powders are fed at room temperature. The condition of the CB-30 mixer is as follows:

20 Mean residence time : 10-18 seconds

Tip speed : 7.5 - 14 m/s

Energy condition : 0.5 - 4 kJ/kg

Mixer speed : 550 - 900 rpm

Jacket temperature : 30°C

25 [Step 2] The agglomerates from the CB-30 mixer are fed to the Schugi FX-160 mixer. 35 kg/hr of neutralized AE₃S liquid (28 % active) is dispersed as finely atomized liquid in the Schugi mixer at about 30-40°C. 20-80 kg/hr of soda ash is added in the Schugi mixer. The condition of the Schugi mixer is as follows:

30 Mean residence time : 0.2 - 5 seconds

Tip speed : 16 - 26 m/s

Energy condition : 0.15 - 2 kJ/kg

Mixer speed : 2000 - 3200 rpm

35 [Step 3] The agglomerates from the Schugi mixer are fed to the KM-600 mixer for further agglomeration, rounding and growth of agglomerates. 60 kg/hr

of ground soda ash (mean particle size of 15 microns) is also added in the KM mixer. Serrated plows are used as mixing elements in the KM mixer. Choppers for the KM mixer can be used to reduce the amount of oversized agglomerates.

The condition of the KM mixer is as follows:

5 Mean residence time : 3- 6 minutes

Energy condition : 0.15 - 2 kj/kg

Mixer speed : 100 - 150 rpm

Jacket temperature: 30 - 40°C

10 The resulting granules from the step 3 have a density of about 700g/l, and can
be optionally subjected to the optional process of cooling, drying, sizing and/or
grinding.

15 Having thus described the invention in detail, it will be obvious to those skilled in the art that various changes may be made without departing from the scope of the invention and the invention is not to be considered limited to what is described in the specification.

What is claimed is:

1. A non-tower process for preparing a granular detergent composition having a density of at least about 600 g/l, comprising the steps of:
 - (a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer wherein conditions of the mixer include (i) from about 2 to about 50 seconds of mean residence time, (ii) from about 4 to about 25 m/s of tip speed, and (iii) from about 0.15 to about 7 kJ/kg of energy condition wherein agglomerates are formed; and
 - (b) spraying finely atomized liquid onto the agglomerates in a mixer wherein conditions of the mixer include (i) from about 0.2 to about 5 seconds of mean residence time, (ii) from about 10 to about 30 m/s of tip speed, and (iii) from about 0.15 to about 5 kJ/kg of energy condition.
2. A non-tower process for preparing a granular detergent composition having a density of at least about 600 g/l, comprising the steps of:
 - (a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer wherein conditions of the mixer include (i) from about 2 to about 50 seconds of mean residence time, (ii) from about 4 to about 25 m/s of tip speed, and (iii) from about 0.15 to about 7 kJ/kg of energy condition, wherein first agglomerates are formed;
 - (b) spraying finely atomized liquid onto the first agglomerates in a mixer wherein conditions of the mixer include (i) from about 0.2 to about 5 seconds of mean residence time, (ii) from about 10 to about 30 m/s of tip speed, and (iii) from about 0.15 to about 5 kJ/kg of energy condition, wherein second agglomerates are formed; and
 - (c) thoroughly mixing the second agglomerates in a mixer wherein conditions of the mixer include (i) from about 0.5 to about 15 minutes of mean residence time and (ii) from about 0.15 to about 7 kJ/kg of energy condition.

3. A process according to claims 1 or 2 wherein said surfactant is selected from the group consisting of anionic surfactant, nonionic surfactant, cationic surfactant, zwitterionic, ampholytic and mixtures thereof.
4. A process according to claims 1 or 2 wherein said surfactant is selected from the group consisting of alkyl benzene sulfonates, alkyl alkoxy sulfates, alkyl ethoxylates, alkyl sulfates, coconut fatty alcohol sulfates and mixtures thereof.
5. The process according to claims 1 or 2 wherein excessive fine powder is formed in step (a), and wherein the excessive fine powder is added to step (b).
6. A process according to claims 1 or 2 wherein an aqueous or non-aqueous polymer solution is dispersed with said surfactant in step (a).
7. A process according to claims 1 or 2 wherein the fine powder is selected from the group consisting of soda ash, powdered sodium tripolyphosphate, hydrated tripolyphosphate, sodium sulphates, aluminosilicates, crystalline layered silicates, phosphates, precipitated silicates, polymers, carbonates, citrates, nitrilotriacetates, powdered surfactants and mixtures thereof.
8. A process according to claims 1 or 2 wherein the finely atomized liquid is selected from the group consisting of liquid silicates, anionic surfactants, cationic surfactants, aqueous polymer solutions, non-aqueous polymer solutions, water and mixtures thereof.
9. A process according to claim 1 wherein the process is a continuous process, wherein the resultant from step (b) is further subjected to cooling and/or drying step, wherein the step creates an internal recycle stream of powder, wherein the internal recycle stream of powder is further added to step (a).

10. A process according to claim 2 wherein the process is a continuous process, wherein the resultant from step (c) is further subjected to cooling and/or drying step, wherein the step creates an internal recycle stream of powder, wherein the internal recycle stream of powder is further added to step (a).
5
11. A granular detergent composition made according to the process of claims 1 or 2.

INTERNATIONAL SEARCH REPORT

Inte: onal Application No

PCT/US 97/09793

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D11/00 C11D17/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|----------|---|-----------------------|
| A | US 4 992 079 A (LUTZ CHARLES W) 12 February 1991 see claims 1,4,5; example I --- | 1,3,4,7, 8,11 |
| A | US 4 169 806 A (DAVIS JERRY E ET AL) 2 October 1979 see examples V,VII --- | 1 |
| A | US 5 554 587 A (CAPECI SCOTT W) 10 September 1996 see column 7, line 9 - line 20 see column 8, line 34 - column 9, line 30; claims 1,6,7,10,11; example I ----- | 1-4,7,8 |

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

* Special categories of cited documents :

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- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
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- *&* document member of the same patent family

Date of the actual completion of the international search

11 September 1997

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INTERNATIONAL SEARCH REPORTInt'l. Application No
PCT/US 97/09793

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|--|------------------|-------------------------|------------------|
| US 4992079 A | 12-02-91 | NONE | |
| US 4169806 A | 02-10-79 | NONE | |
| US 5554587 A | 10-09-96 | WO 9707196 A | 27-02-97 |